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(71) Applicant: **HERCULES INCORPORATED**
Hercules Plaza
Wilmington Delaware 19894(US)

(72) Inventor: **Schmalz, Alfred Chandler**
2594 Harvest Drive
Conyers, Georgia 30208(US)

(74) Representative: **Lederer, Franz, Dr. et al**
Lederer, Keller & Riederer, Patentanwälte,
Lucile-Grahn-Strasse 22 22
D-8000 München 80(DE)

(54) **Rewettable polyolefin fiber and corresponding nonwovens.**

(57) A method for imparting hydrophilic properties to non-woven material containing hydrophobic polyolefin-containing fiber or fibrillated film by applying onto the surface of the fiber or fibrillated film an aqueous alkoxylated surfactant composition comprising at least 80% of alkoxylated or alkoxylated and hydrogenated triglyceryl esters of 18-carbon fatty acids including a major portion of alkoxylated ricinolein or alkoxylated and hydrogenated ricinolein, or a water-soluble polyalkoxylated polydimethylsiloxane combined with an antistatic compound, or 0.5 to 80% of the said alkoxylated ricinolein or alkoxylated and hydrogenated ricinolein and 20 to 99.5% by weight of a water-soluble polyalkylene modified polydimethylsiloxane combined with the antistatic compound, and forming the fiber or fibrillated film into a nonwoven material.

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REWETTABLE POLYOLEFIN FIBER AND CORRESPONDING NONWOVENS

This invention relates to a method for imparting hydrophilic properties to nonwoven material containing hydrophobic fiber or fibrillated film by applying onto the surface of the fiber or fibrillated film an aqueous alkoxylated surfactant composition, and to such nonwoven material to which hydrophilic properties have been imparted.

Products used for personal hygiene, such as catamenial devices, disposable diapers, incontinence pads and the like, frequently have a fluid-absorbent core, usually comprising one or more layers of absorbent material, a facing or cover stock layer of essentially nonabsorbent material that encloses the absorbent core and prevents skin contact with the core, thus tending to isolate any fluids already absorbed in the core, and a fluid impervious barrier sheet to protect the wearer's clothing from stain or wetting by any absorbed fluids.

On the side that is placed against the body, the facing or cover stock material should be pervious to fluids with minimal surface fluid retention, so as to promote the immediate transfer of the fluid into the absorbent core material and protect the wearer from contact with the previously wetted absorbent material. It should also inhibit lateral migration of fluid along its surface, and feel smooth and soft to the touch. It may have additional characteristics that are sometimes desired, such as visual opacity, particular coloring, and a lustrous outer surface.

To obtain such desirable characteristics, it is recognized that the cover, stock must comprise essentially hydrophobic polymeric material, such as polyolefin fiber or film, that is sufficiently hydrophilic to instantly receive and transmit aqueous fluids. However, the material should also maintain that ability even after several wettings (sometimes colloquially referred to in the art as "insults"); an objective that is much more difficult to achieve. Thus agents used to promote such hydrophilicity must have the ability to resist the inherent tendency of such wettings to reduce their effectiveness by leaching or "wash-out". This is particularly important in the case of cover stock for diapers, so as to avoid lateral migration of liquid and leakage at the edges after one wetting. Also, of course, treatment with such agents should not interfere with fabric-bonding steps in the manufacture of the final product and its wet strength.

U.S. Patent 4,578,414 discloses a method for imparting surface wettability to hydrophobic polyolefin fibers, including polypropylene, that includes compounding with the bulk molten polymer a surface-active agent that comprises an alkoxylated alkylphenol and/or a polyoxyalkylene fatty acid ester, or either of them together with a triglyceride. U.S. Patent 3,853,601 discloses a process for making a microporous polypropylene film hydrophilic by coating it with polyoxyethylene polymethyl siloxane, to enhance its short-term wettability when disposed in an electrolytic cell filled with a strong electrolyte.

Japanese Patent 63211369 discloses the treatment of nonwoven synthetic fiber including polypropylene with a polysiloxane-polyoxyethylene copolymer to provide a durable hydrophilic finish on water-absorptive nonwoven top sheets for diapers and the like.

However, there is still a need for methods that impart greater resistance to wash-out and leaching by repetitive wettings, that possess better fluid control, and reduce interference with bonding properties under high speed commercial operation.

According to the invention, a method for imparting hydrophilic properties to nonwoven material containing hydrophobic polyolefin-containing fiber or fibrillated film by applying onto the surface of the fiber or fibrillated film an aqueous alkoxylated surfactant composition, is characterized in that the surfactant composition comprises at least 80% of alkoxylated or alkoxylated and hydrogenated triglyceryl esters of 18-carbon fatty acids including a major portion of alkoxylated ricinolein or alkoxylated and hydrogenated ricinolein, or a water soluble polyalkoxylated polydimethylsiloxane combined with an antistatic compound, or 0.5 to 80% of the said alkoxylated ricinolein or alkoxylated and hydrogenated ricinolein and 20 to 99.5% by weight of a water-soluble polyalkylene-modified polydimethylsiloxane combined with the antistatic compound, the amount of the surfactant composition being about 0.2% to 2% by weight of the fiber or fibrillated film, and forming the fiber or fibrillated film into the nonwoven material.

Preferably, the surfactant composition comprises a water-soluble ethoxylate of polydimethylsiloxane such as that commercially obtainable from Union Carbide Corporation as Y-12230 combined with 0.1% to 0.3% by wt. of an antistatic agent such as a neutralized ester of phosphoric acid and alcohol (for instance obtainable commercially as Lurol AS-Y from G.A. Goulston Company) or any of the similar known phosphate-based antistatic agents including alkoxylated phosphates, potassium salts, amine salts, and alkoxylated amine salts. The surfactant composition obtainable from Union Carbide Corporation as Y-12230 may be combined with up to 50% of an equivalent polyalkylene oxide-modified polydimethyl siloxane also obtainable from Union Carbide Corporation under the trademark Silwet, such as Silwet 7603.

The alkoxylated mixture of triglycerides of fatty acids that comprise least 80% of the surfactant

composition can be conveniently obtained by conventionally esterifying and alkoxylation, and if desired hydrogenating, castor oil. The said mixture of triglyceride fatty acids will in that case include approximately 87% of ricinoleic, and approximately 7% of oleic, 3% of linoleic, 2% of palmitic, and 1% of stearic acids, the alkoxylation preferably being by conventional treatment with polyoxyethylene. Other sources may provide a different mixture, for example, one including more fatty acid containing 18-carbon atoms, such as oleic, linoleic, and stearic acids.

Also preferably, for convenience, the surfactant composition comprises ethoxylated castor oil in liquid form commercially obtainable from Henkel A.G. as Dacospin® 1735A, or the fiber lubricant emulsifier also obtainable in liquid form from Henkel A.G. as Stantex® A241, or the hydrogenated castor oil in liquid form commercially obtainable from Henkel A.G. as Emery® 32148 or 32149. The surfactant composition may comprise the said alkoxylation (preferably ethoxylated) mixture of triglycerides of fatty acids, in combination with 20% to 99.5% by weight of the water-soluble ethoxylate of polydimethylsiloxane such as that commercially obtainable from Union Carbide Corporation as Y-12230.

Preferably, the amount of the surfactant composition is from 0.5% to 1% by weight of the fiber or fibrillated film.

The surfactant composition may be applied to continuous spun fibers or filaments or fibrillated film, by conventionally drawing them over a feed or "kiss" roll partially immersed in a bath of the surfactant composition, or by dipping them in a bath, or by spraying with the liquid, and drying them.

The fiber or films used to form webs and nonwovens, as above described, are preferably spun or cast from isotactic polypropylene or art-recognized hydrophobic copolymers thereof and/or fixtures thereof, the spin melt conveniently having a weight average varying from about 3×10^5 to about 5×10^5 , a molecular weight distribution of about 5.0-8.0, a melt flow rate of about 2.5 to about 4.0 g/10 min., plus a spin temperature conveniently within a range of about 220°C.-300°C.

The webs used to form the nonwoven materials can of course be conventionally formed by the well-known bonding techniques used to form nonwoven materials from fiber or fibrillated film, for instance using adhesive binders, heated calender rolls, or needle punching.

The method according to the invention does not interfere with the use of additives conventionally incorporated in the spin melt of polyolefin-containing resin or topically applied to the fiber or fibrillated film, including pH stabilizers such as calcium stearate, antioxidants, degrading agents, pigments, including whiteners and colorants such as TiO_2 . Generally such additives individually vary in amount from about 0.1% to 3% by weight of the treated material.

The following examples further illustrate, but do not limit the present invention. The following tests were performed and the results reported in the tables:

"Sink time" (liquid absorbency time): Five (5) gram samples of each filament are loosely packed into identical 3 gram mesh baskets (in accordance with ASTM Method D-1117-79), increases in sink time or submergence time after repeated treatments representing the loss of hydrophilicity.

"Strike-through time" is the time in seconds required for 5 ml of syn-urine to pass through a single sheet of nonwoven fabric then into absorbent paper (filter paper) pads.

"Strike-through time/rewet" or "Strike Time Rewets" is performed by first carrying out the Strike-through time test with 5 ml of liquid and fresh absorbent paper and then measuring the times for successive additions of 10 ml of the same liquid to pass through the fabric; the time in seconds is recorded in the indicated column. After each addition, the value in the "Rewets" column is determined by placing an absorbent pad on top of the fabric and under a 3.63 kg (8 lb) weight, and measuring the weight of liquid in grams that is passed back during 5 minutes from the wet pad through the fabric into the top pad. As already indicated, each wetting is referred to as an "Insult".

The invention is further illustrated, but not limited, by the following Example and Tables:

EXAMPLE 1

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A. Two batches of isotactic polypropylene are fed through a 1 1/2" extruder and conventionally spun, using a 210 hole spinnerette at 285°C., air quenched, and resulting continuous 2.5 dpf and 310 dpf batch filaments passed over a feed or kiss roll partly immersed in a tank of modifier composition comprising ethoxylated polydimethylsiloxane obtained commercially from Union Carbide as "Y-12230" together with about 1% by weight of Lurol AS-Y obtained commercially from G.A. Goulston Incorporated; two batches are prepared varying in duration and speed so as to topically apply 0.87 wt. % and 0.36 wt. % of the modifier composition respectively. The resulting spin yarn is drawn, passed through a crimper, topically treated with

finish, chopped to 1.5" staple, then carded into webs weighing about 20 g/yd², and routinely calendar-bonded at 165° C. The respective test nonwoven materials are cut into test strips identified as S-1, S-2 and S-3 for conventional strike through and rewet tests using Syn-urine™ (an aqueous commercial product obtained from Jayco Pharmaceutical Company of Camp Hill, PA) as the wetting fluid. Test results are reported in Table I below. An average of several 2.5 dpf control samples (C-1) are identically prepared, except for the absence of topically applied modifier composition, and the corresponding nonwoven tested and reported in Table I.

TABLE I

THERMAL BONDED FABRIC						
TOPICAL TREATMENT						
				Insults		
				Strike-Through		
<u>Samples</u>	<u>Denier</u>	<u>Finish</u>	<u>Level</u>	<u>Rewet</u>	<u>Time (Sec)</u>	<u>Rewets</u>
	(dpf)					
S-1	2.5	Y-12230/0.5% ASY	0.87%	1	1.2	0.11
			0.87%	2	1.1	0.10
			0.87%	3	1.2	0.10
			0.87%	4	1.8	0.11
			0.87%	5	2.4	0.11
S-2	3.0	Y-12230/0.5% ASY	0.36%	1	1.0(*1)	0.11
			0.36%	2	178.5	0.11
			0.36%	3	56.3	0.11
			0.36%	4	108.3	0.11
			0.36%	5	15.4	0.10
S-3	2.5	Y-12230/0.5% ASY	.34	1	1.3	.16
			.34	2	21.8	.13
			.34	3	20.3	.13
			.34	4	28.1	.13
			.34	5	152.4	.12
C-1	2.5	No Modifier	0	1	1.6	.10
		No Modifier	0	2	300	.10

*1 Inconsistent results believed due to contaminated spin lubricant.

B. 3 dpf spun fiber is conventionally prepared by batch, using polypropylene fiber and a spinning device as described in Example 1A, to which

1. 50% Y12230/50% Silwet® 7603, or
2. Dacospin® and 1735A, or
3. Stantex® A241

are respectively topically applied using a kiss wheel, and the treated fiber air dried as before. Five (5) gram samples of 1.5 inch uncrimped staple fiber from each batch are loosely packed into identical 3 gram mesh baskets for sink-time tests in accordance with ASTM Method D-1117-79, whereby an increase in sink time (i.e., increase in time of submergence) after repeated insults by Syn-urine is interpreted as the result of a wash out or leach out applied of wetting agent and corresponding loss in desired hydrophilic properties. Test results are reported in Table 2 as Samples S-4, S-5, and S-6 and the corresponding control, having 5 gm of the spun polypropylene without modified composition, is reported as C-3 in Table 2.

TABLE 2

REWETTABLE POLYPROPYLENE SPIN YARN					
TOPICAL TREATMENT					
Samples	Fiber	Type Finish	Modifier Composition	Insults	Sink Time (Sec)
	(dpf)				
S-4	3.0	50% Y12230 50% Silwet 7603	2.0%	1 2 3 4 5	1 1 3 2 3
S-5	3.0	Dacospin 1735A	1.0%	1 2 3 4 5	2 7 10 22 34
S-6	3.0	Stantex A241	1.6%	1 2 3 4 5	2 15 15 14 10
C-3	2.5	---	---	1 2 3 4	1.1 4.0 60.0 600.0

Claims

1. A method for imparting hydrophilic properties to nonwoven material containing hydrophobic polyolefin-containing fiber or fibrillated film by applying onto the surface of the fiber or fibrillated film an aqueous alkoxyated surfactant composition, characterized in that the surfactant composition comprises at least 80% of alkoxyated or alkoxyated and hydrogenated triglyceryl esters of 18-carbon fatty acids including a major portion of alkoxyated ricinolein or alkoxyated and hydrogenated ricinolein, or a water-soluble polyalkoxyated polydimethylsiloxane combined with an antistatic compound, or 0.5 to 80% of the said alkoxyated ricinolein or alkoxyated and hydrogenated ricinolein and 20 to 9915% by weight of a water soluble polyalkylene modified polydimethylsiloxane combined with the antistatic compound, the amount of the surfactant composition being about 0.2% to 2% by weight of the fiber or fibrillated film, and forming the fiber or fibrillated film into the nonwoven material.
2. A method for imparting hydrophilic properties to nonwoven material as claimed in claim 1, further characterized in that the polyalkoxyated polydimethylsiloxane is a polyethoxyated polydimethylsiloxane.
3. A method for imparting hydrophilic properties to nonwoven material as claimed in claim 2, further characterized in that the antistatic agent is a phosphate-based antistatic agent.
4. A method for imparting hydrophilic properties to nonwoven material as claimed in claim 3, further characterized in that the antistatic agent is a neutralized ester of phosphoric acid and alcohol.
5. A method for imparting hydrophilic properties to nonwoven material as claimed in claim 1, further characterized in that the surfactant composition comprises an alkoxyated mixture of fatty acids including approximately 87% of ricinoleic acid, or a hydrogenated derivative of the said mixture.
6. A method for imparting hydrophilic properties to nonwoven material as claimed in either of claims 1 or 5, further characterized in that the mixture of triglyceride fatty acids is ethoxyated.
7. A method for imparting hydrophilic properties to nonwoven material as claimed in any of the preceding claims, further characterized in that the amount of the surfactant composition is about 0.2% to 2% by

weight of the fiber or fibrillated film.

8. A method for imparting hydrophilic properties to nonwoven material as claimed in any of the preceding claims, further characterized in that the amount of the antistatic agent is 0.11% to 0.3% by weight of the fiber or fibrillated film.

5 9. Use of polyethoxylated polydimethylsiloxane for imparting hydrophilic properties to nonwoven material containing hydrophobic polyolefin-containing fiber or fibrillated film by applying it in an aqueous medium onto the surface of the fiber or fibrillated film together with a phosphate-based antistatic agent.

10 10. Use of an alkoxyated mixture of fatty acids including approximately 87% of ricinoleic acid, or a hydrogenated derivative of the said mixture for imparting hydrophilic properties to nonwoven material containing hydrophobic polyolefin-containing fiber or fibrillated film by applying it in an aqueous medium onto the surface of the fiber or fibrillated film.

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EUROPEAN SEARCH REPORT

Application Number

DOCUMENTS CONSIDERED TO BE RELEVANT			EP 90114492.3
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 5)
D,X	<u>US - A - 4 578 414</u> (SAWYER) * Claims; column 4, lines 60-63; column 6, lines 23-41 *	1,5-7 10	D 06 M 13/224 D 06 M 15/643 D 06 M 13/292 //D 06 M 101:20 A 61 F 13/46
D,X	<u>US - A - 3 853 601</u> (TASKIER) * Claims *	1,2,7 9	
X	<u>EP - A2 - 0 325 543</u> (JAMES RIVER CORPORATION OF VIRGINIA) * Totality *	1	
D,X	DERWENT ACCESSION NO. 88-289 120, Questel Tele-systems (WPIL) DERWENT PUBLICATIONS LTD., London * Abstract * & JP-A-63 211 369 (MITSUI PETROCHEM)	1,2,9	TECHNICAL FIELDS SEARCHED (Int. Cl. 5) D 06 M A 61 F D 04 H
Y	<u>FR - A1 - 2 341 691</u> (BAYER AKTIENGESELLSCHAFT) * Claims; page 5, lines 9-15 *	1,7,8	
Y	<u>US - A - 4 361 611</u> (SCHÄFER) * Claims; column 2, lines 30-33 *	1,3,4, 8	
A	<u>US - A - 3 968 042</u> (ERICKSON) * Claims *	1,2,9	
The present search report has been drawn up for all claims			
Place of search VIENNA		Date of completion of the search 12-11-1990	Examiner SCHÄFER
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			

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